OIPE JUN 0 9 2003 PATENT & TRADEMARK OFFICE

#9 (NE) Reconsideration w/ attach SDams 6/12/03

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the Application of: **KANO, Takashi et al.**

Group Art Unit: **2812**

Serial No.: **09/941,982**

Examiner: **MULPURI, Savitri**

Filed: **August 30, 2001**

P.T.O. Confirmation No.: 7536

For: **METHOD OF FORMING NITRIDE-BASED SEMICONDUCTOR LAYER, AND METHOD OF MANUFACTURING NITRIDE-BASED SEMICONDUCTOR DEVICE**

RECEIVED JUN 11 2003 TECHNOLOGY CENTER 2800

REQUEST FOR RECONSIDERATION AFTER FINAL REJECTION UNDER 37 CFR §1.116
- EXPEDITED RESPONSE -
GROUP ART UNIT 2812

MAILSTOP AF

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

June 9, 2003

Sir:

In response to the Office Action dated **January 8, 2003**, Applicants request favorable reconsideration of the above-identified application. Claims 1-5, 7-14 and 16-18 are pending.

Claims 1, 3, 5, 7-10, 12, 14, 16 and 18[1] were rejected under 35 USC §102(b) as being anticipated by Imai et al. In addition, the Examiner has repeated the rejection of claims 2, 4, 11 and 13 under 35 USC §103(a) as being unpatentable over Imai et al. and the rejection of claim 17 under 35 USC §103(a) as being unpatentable over Kondow et al. in combination with Imai et al.

In the Examiner's Response to Arguments, the Examiner argues that Imai et al. teaches broad

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[1] The Examiner included claims 6 and 15 in the rejection, but such clearly is a typographical error since these claims have been canceled.

ranges for both growth rate and thickness. The Examiner also comments on the description of the comparative example as being irrelevant, and argues that the combination of the specific claimed thickness and growth rate would have been inherently taught by Imai et al.

Although Imai et al. provides broad disclosures of both thickness and growth rate, Imai et al. fails to teach the specific claimed combination of thickness and growth rate. Furthermore, such a combination would not have been inherently taught by Imai et al. as asserted by the Examiner.

Such should be clear from a review of all the working examples of Imai et al. which show that there is no suggestion of growing a buffer layer at a growth rate of at least 7 Å/sec to have a thickness of 50Å to 300Å. Examples 4 and 5 provide the fastest growth rate of the buffer layer of 1.5 Å/sec. The remaining working examples of Imai et al. show slower growth rates.

Comparative Example 5 of Imai et al. was highlighted in the prior response since it disclosed a growth rate of 150 Å/sec, producing a first layer having a thickness of more than 5000 Å. Contrary to the assertion of the Examiner, this example is relevant as part of the disclosure of Imai et al. in showing what Imai et al. actual teaches.

Imai et al. discloses that the thickness of the layer depends on the film growth rate (see column 7, lines 6-8). Contrary to the assertion of the Examiner, a growth rate of at least 7 Å/sec in combination with a thickness of 50-300 Å is not inherently taught by Imai et al. Thus, the disclosure rate by Imai et al. that the thickness of the layer depends on the film growth does not teach or suggest that growing a layer at a relatively fast growth rate (of at least 7 Å/sec) would grow the buffer layer to have a film thickness in the range from 50Å to 300Å.

The disclosure of Imai et al. that the thickness of the layer depends on the film growth rate is further understood from its examples.

Imai et al. specifically describes the following growth rates and thicknesses:

- growth rate of 0.6Å/sec. for thickness of 500 to 700 Å (Examples 13 and 14)
- growth rate of 1.2Å/sec. for thickness of 1000 to 4600 Å (Examples 1, 2 and 6 to 12)
- growth rate of 1.3Å/sec. for thickness of 1700 Å (Example 3)
- growth rate of 1.5Å/sec. for thickness of 1700 to 1900 Å (Examples 4 and 5)
- growth rate of 150 Å/sec. for thickness of 5000 Å (Comparative Example 5)

It is understood from the relation between the growth rates and the thicknesses specifically described in Imai et al. that a layer having a small thickness is formed at a low growth rate and a layer having a large thickness is formed at a high growth rate.

In other words, it is expected from the relation between the growth rates and the thicknesses described in Imai et al. that the growth rate is reduced in order to form a layer having an extremely small thickness. However, it is not obvious to form such a thin layer at an extremely high growth rate of at least 7 Å/sec. as claimed in the claims of the present application.

As to the effects brought out from the relation between the growth rate and the thickness defined in the present invention, applicants present the contents of a report at the meeting of the Japan Society of Applied Physics, September 2000.

At this meeting, Kano et al, the inventors, reported the effects brought out from the relation between the growth rate and the thickness defined in the present invention with the title "Quality Improvement of GaN Layer Employing Fast-Grown Low-Temperature AlGaN Buffer Layer" on September 5, 2000. Copies of Extended Abstracts (The 61st Autumn Meeting, 2000) (5P-Y-1) and the OHP sheets used for the report are attached.

The eighth and ninth OHP sheets show sectional TEM photographs of a sample prepared by forming an AlGaN buffer layer having a thickness of 120 Å on a sapphire substrate at a growth rate of 6.7 Å/sec. and forming a GaN layer thereon, and sectional TEM photographs of a sample prepared by forming an AlGaN buffer layer having a thickness of 120 Å on a sapphire substrate at a growth rate of 25.0 Å/sec. and forming a GaN layer thereon, respectively. The eighth OHP sheet (300,000 magnification) proves that the sample prepared at the growth rate of 25.0 Å/sec. in the range claimed in claims of the present application has a smaller number of upwardly extending defects as compared with the sample prepared at the growth rate of 6.7 Å/sec. out of the range claimed in claims of the present application. The ninth OHP sheet (2,000,000 magnification) proves that a trapezoidal portion (a white portion) is formed on the AlGaN buffer layer in the sample prepared at the growth rate of 25.0 Å/sec. in the range defined in the present invention. As shown in the tenth OHP sheet, defects transversely bend without extending upward on the upper surface of the trapezoidal portion in the sample prepared at the growth rate of 25.0 Å/sec. in the range defined in the present invention, to attain an unexpected remarkable effect.

The contents of this report are described from page 19, line 1 to page 21, line 19 of the English specification of the present application. The results of measurement of X-ray half widths (FWHM) shown in Fig. 2 indicate that the range of the growth rate defined in the present invention, i.e., at least 7 Å/sec., is the critical range for attaining such an unexpected effect.

Accordingly, when considering the entire disclosure of Imai et al., even based on the separate broad disclosures of film growth rate and thickness, Imai et al. does not provide any teaching of the combination of a growth rate of at least 7 Å/sec to grow a layer having a film thickness from 50 Å to 300 Å.

Applicants are considering the preparation of a Declaration demonstrating the unexpected results of the present invention in the event that the Examiner does not consider the present response as placing the application in condition for allowance.

For at least the foregoing reasons, the claimed invention distinguishes over the cited art and defines patentable subject matter. Favorable reconsideration is earnestly solicited.

Should the Examiner deem that any further action by applicants would be desirable to place the application in condition for allowance, the Examiner is encouraged to telephone Applicants' undersigned attorney.

U.S. Patent Application Serial No. 09/941,982

In the event that this paper is not timely filed, Applicants respectfully petition for an appropriate extension of time. Please charge any fees for such an extension of time and any other fees which may be due with respect to this paper, to Deposit Account No. 01-2340.

Respectfully submitted,

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23850
PATENT TRADEMARK OFFICE

Attachment: Petition for Extension of Time
Partial Translations of Extended Abstracts (the 61st Autumn Meeting, 2000)
Translation of OHP sheets used in the 61st Autumn Meeting, 2000 of the Japan Society of Applied Physics

Q:\FLOATERS\SGA\01\011083\REQUEST FOR RECONSIDERATION

Part of #9

Partial Translation of Extended Abstracts
(The 61st Autumn Meeting, 2000);
The Japan Society of Applied Physics

5p-Y-1

High quality GaN film on low-temperature AlGaN buffer layer grown with high growth rate

Sanyo Electric Co., Ltd.

Microelectronics Research Center

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1. Introduction A GaN layer on sapphire is generally grown on a buffer layer grown at a low temperature, and it is important to optimize conditions of the buffer layer and the GaN layer grown thereon for improving the characteristics of a nitride-based light-emitting device. This time we have found out that a high-quality GaN film can be obtained by remarkably increasing the growth rate for a buffer layer, and report this.

2. Experiment GaN was grown on c-plane sapphire by atmospheric pressure MOCVD in a two-step growth method. A buffer layer was prepared from AlGaN, and growth temperatures for the buffer layer and the GaN layer grown thereon were 600°C and 1080°C respectively. The growth rate for the buffer layer

was varied for evaluating the X-ray diffraction FWHM, surface morphology etc.

3. Conclusion Fig. 1 shows the relation between the growth rate for the AlGaN buffer layer and the X-ray diffraction FWHM. The X-ray diffraction FWHM was reduced as the growth rate was increased, and an excellent value of 248 arc sec. was obtained when the growth rate for the low-temperature AlGaN buffer layer was 25 Å/sec. (9 μm/h). The surface morphology was a mirror surface at this time, as shown in Fig. 2.

Fig. 1 Relation Between Growth Rate of AlGaN Low-Temperature Buffer Layer and Full Width at Half Maximum of X-ray of GaN Layer

Fig. 2 Surface Morphology

USAF Catalog Number : AP001130-01

2000年（平成12年）秋 季

第61回応用物理学会学術講演会
講 演 予 稿 集

Extended Abstracts (The 61st Autumn Meeting, 2000);
The Japan Society of Applied Physics

No. 1

- 1 放射線・プラズマエレクトロニクス
  Radiation・Plasma Electronics
- 2 計測・制御
  Measurement and Control
- 8 応用物性
  Applied Material Physics
- 9 超伝導
  Superconductivity
- 13 結晶工学
  Crystal Engineering
- 15 応用物理一般
  General Applied Physics
- 合同セッションC：「Siナノ・デバイス」
  Si Nano-Devices
- 合同セッションD：「プラズマCVDの基礎と（デバイス）応用」
  Fundamental Science of plasma CVD and its application

期 日：2000年9月3日（日）～7日（木）

第１分冊　講演分科日程表

北海道工業大学

大分類分科名 中分類分科名	9月3日（日）午前	9月3日（日）午後	9月4日（月）午前	9月4日（月）午後	9月5日（火）午前	9月5日（火）午後	9月6日（水）午前	9月6日（水）午後	9月7日（木）午前	9月7日（木）午後
1．放射線・プラズマエレクトロニクス										
1 放射線・加速器・原子炉					3-V 67 9:30-12:30		3-V 70 9:30-12:30	3-V 74 13:30-17:00		
2 プラズマプロセスの基礎			2-F 78 9:00-12:00		2-F 82 9:00-12:00	2-F 86 13:00-17:30	2-F 91 9:00-12:00	2-F 95 13:00-17:15	2-F 100 9:00-12:00	2-F 104 13:00-15:00
3 放電・プラズマ現象一般					7-ZM （ショート） 10:30-12:40	→ポスター 107 15:30-17:30				
2．計測・制御										
1 計測・制御								図-A 118 13:00-16:45		
2 測定標準・精密計測							図-A 115 9:00-12:00			
3 情報処理応用計測										
8．応用物性										
1 磁性材料・磁性体	3-V 125 9:30-12:30	3-V（後に中分類8.2） 132 13:30-16:45								
2 磁気応用デバイス		3-V（前に中分類8.1） 128 16:45-17:15								
3 誘電材料・誘電体							P10 133 9:30-11:30			
4 微粒子・粉体	P2 138 9:30-11:30									
5 単一電子輸送現象	3-T 140 9:30-13:00									
6 熱電変換	1-R 144 9:00-12:00		1-M 148 9:00-11:45	1-M 151 13:00-15:00						
7 新機能材料・新物性		3-T 154 13:30-17:00								
9．超伝導										
1 基礎物性	7-ZL 159 10:00-13:00	7-ZL 162 14:00-18:15	7-ZL 168 10:00-12:45	7-ZL 171 14:00-15:45						
2 磁束ピンニング，Jc					7-ZL 173 10:00-12:45	7-ZL 177 14:00-15:45				
3 線材，バルク・薄膜作製									7-ZL 179 9:00-12:00	7-ZL 183 13:00-14:45
4 薄膜及び接合作製			5-ZK（ショート） 10:00-12:00	→ポスター 185 15:30-17:00	2-L（ショート） 9:00-11:10	→ポスター 193 13:00-15:00				
5 アナログ応用と関連技術	7-ZM 202 10:00-13:00	7-ZM 205 14:00-18:30	7-ZM 211 10:00-12:45							
6 デジタル応用と回路プロセス							7-ZL 214 10:00-12:45	7-ZL 218 14:00-16:15		
13．結晶工学										
1 バルク結晶成長	3-Z 221 9:30-12:30	3-Z 224 13:30-18:15								
2 II-VI族エピ結晶			3-Z 230 9:45-12:15	3-Z 233 13:30-17:00	3-Z 238 9:30-12:15	3-Z 241 13:30-17:45	3-Z 246 9:30-12:30			
3 III-V族エピ結晶	3-ZA 250 9:30-12:30	3-ZA 254 13:30-18:00	3-ZA 259 9:30-12:30	3-ZA 263 13:30-17:00	3-ZA 267 9:30-12:30	3-ZA 271 13:30-18:00				
4 III-V族窒化物結晶	3-Y（ショート） 9:30-12:30	→ポスター 277 13:00-15:00	3-Y（ショート） 9:30-12:30	→ポスター 288 13:00-15:00	3-Y 299 9:30-12:30	3-Y 302 13:30-18:00			2-H 317 9:00-12:00	
							2-L 308 9:30-12:30	2-L 312 ※13:30-17:50	2-L 320 9:00-12:00	2-L 324 13:00-15:30
5 IV族，IV-IV族結晶	3-ZB 327 9:30-12:30	3-ZB 331 13:30-18:30	3-ZB 337 9:30-12:30	3-ZB 341 13:30-16:45	3-ZB 345 9:30-12:30	3-ZB 349 13:30-17:30				
6 エピタキシーの基礎								3-ZA 354 13:30-16:00		
7 結晶評価・素子技術								3-Z 357 13:30-17:15	3-Z 362 9:00-12:00	3-Z 365 13:00-15:00
8 欠陥物理・結晶改質							3-ZA 368 9:30-12:30			
15．応用物理一般										
1 応用物理一般						2-D 373 ※13:10-15:30				
2 物理教育			P5 375 9:30-11:30							
3 新技術（センサ，電気化学等）							7-ZQ 380 10:00-13:00	7-ZQ 384 14:00-16:15		
4 トライボロジー（変換素子等）					3-T 386 10:30-11:30					
5 エネルギー変換・貯蔵					7-ZN（前に中分類15.6） 388 12:30-13:00					
6 資源・環境					7-ZN（後に中分類15.5） 391 10:00-12:30					
7 磁場応用			7-ZQ 391 9:30-12:15	7-ZQ 395 13:15-17:00						

中分類分科名
1.1 放射線・加速器・原子炉
1.2 プラズマプロセスの基礎（反応性プラズマの生成、物性、気相・表面反応過程、診断、装置など）
1.3 放電・プラズマ現象一般（衝突・放射過程、輸送過程、光源・ディスプレイ・気体レーザーなどの応用）
2.1 計測・制御（計測手法、測定装置・システム・センサの開発、機器分析、デバイスなど）
2.2 測定標準・精密計測（測定標準、基礎物理定数、物性定数、単位、微小領域など）
2.3 情報処理応用計測（インテリジェント計測、データ処理、パターン認識、情報処理、カオスなど）
8.1 磁性材料・磁性体
8.2 磁気応用デバイス
8.3 誘電材料・誘電体（強誘電体）
8.4 微粒子・粉体
8.5 単一電子輸送現象
8.6 熱電変換
8.7 新機能材料・新物性（金属、セラミックス、低温物性など）
9.1 基礎物性
9.2 磁束ピンニング，Jc
9.3 線材、バルク及び厚膜作製プロセス
9.4 薄膜及び接合作製プロセス
9.5 アナログ応用と関連技術
9.6 デジタル応用と回路プロセス
13.1 バルク結晶成長
13.2 II-VI族エピタキシャル結晶
13.3 III-V族エピタキシャル結晶
13.4 III-V族窒化物結晶
13.5 IV族、IV-IV族結晶（Siエピ、SiGe、SiC、ダイヤモンドなど）
13.6 エピタキシーの基礎（機構、理論・シミュレーション、成長法）
13.7 結晶評価・素子技術
13.8 欠陥物理・結晶改質
15.1 応用物理一般（熱、音響、超音波、液体、静電気など）
15.2 物理教育
15.3 新技術（センサー、電気化学、人間工学など）
15.4 トライボロジー（変換素子、マイクロマシンなど）
15.5 エネルギー変換・貯蔵
15.6 資源・環境
15.7 磁場応用（磁場効果、磁気エネルギー、磁場配向、磁気科学、磁場中計測、強磁場）

1 放射線・プラズマエレクトロニクス
Radiation・Plasma Electronics

2 計測・制御
Measurement and Control

8 応用物性
Applied Material Physics

9 超伝導
Superconductivity

13 結晶工学
Crystal Engineering

15 応用物理一般
General Applied Physics

合同セッションC (Joint Session C)
「Siナノ・デバイス」
Si Nano-Devices

合同セッションD (Joint Session D)
「プラズマCVDの基礎と（デバイス）応用」
Fundamental Science of plasma CVD and its application

13 結晶工学

Crystal Engineering

		講演番号	予稿掲載頁
13.1	バルク結晶成長 Bulk Crystal Growth	3a-Z-1～3p-Z-18	221～230
13.2	II-VI族エピタキシャル結晶 II-VI Epitaxial Growth	4a-Z-1～4p-Z-13 5a-Z-1～5p-Z-16 6a-Z-1～11	230～237 238～246 246～250
13.3	III-V族エピタキシャル結晶 III-V Epitaxial Growth	3a-ZA-1～3p-ZA-17 4a-ZA-1～4p-ZA-13 5a-ZA-1～5p-ZA-17	250～259 259～267 267～276
13.4	III-V族窒化物結晶 III-V Nitride Epitaxial Growth	3a-Y-1～33 4a-Y-1～33 5a-Y-1～5p-Y-17 6a-L-1～6p-L-16 7a-H-1～11 7a-L-1～7p-L-10	277～287 288～298 299～308 308～316 317～320 320～327
13.5	IV族、IV-IV族結晶 IV and IV-IV Epitaxial Growth	3a-ZB-1～3p-ZB-19 4a-ZB-1～4p-ZB-12 5a-ZB-1～5p-ZB-15	327～337 337～345 345～353
13.6	エピタキシーの基礎 Fundamental of Epitaxy	6p-ZA-1～10	354～357
13.7	結晶評価・素子技術 Characterization	6p-Z-1～14 7a-Z-1～7p-Z-8	357～361 362～368
13.8	欠陥物理・結晶改質 Physics and Engineering of Defects	6a-ZA-1～11	368～371

5a－Y－10

ナノコラムGaN上へのGaNのオーバーグロースによる残留歪みの低減化

Reduction of residual strain in overgrown GaN on nano-columnar GaN

上智大学　理工学部　*草部一秀, 山田隆之, 豊浦洋祐, 坂内亮, 菊池昭彦, 岸野克巳
Sophia University　*K.Kusakabe, T.Yamada, Y.Toyoura, R.Bannai, A.Kikuchi, K.Kishino
E-mail : kusakabe@katsumi.ee.sophia.ac.jp

はじめに　一般にGaNは異種基板へのヘテロ成長が行われるが、格子不整合や熱膨張係数の違いに起因する残留歪みが成長層に存在する。この残留歪みはピエゾ分極を誘起しその結果として、光学特性やキャリアの輸送特性に多大な影響を与えることが知られている。我々は再成長や成長後のプロセス無しで、サファイア基板上に直接成長したフリースタンディングのGaNを得る方法を提案する。

実験および結果　フリースタンディングGaNの成長は、RF-plasma窒素を用いたRF-MBE法により行った。成長基板は前処理によって原子層ステップを形成させたc面サファイアを用いた。基板窒化後、成長温度850°CにてAlN bufferを成長し、窒素過剰下でGaNナノコラム[1]の成長を行った。ナノコラムの直径は100nm程度でありエア・ブリッジ構造を有することから、バッファ層として採用することで2軸性歪みを緩和することが期待される。引き続き、成長温度750°CにてGaN:Siのオーバーグロース層を2.9μm成長させた。ナノコラムの3次元成長から2次元成長に推移した結果として、平坦な表面のGaN層が得られた(Fig.1)。4結晶X回折測定より、サンプルの残留歪みを評価したところ膜厚2.9μmにも関わらず、フリースタンディングの場合のc軸長=5.185Åを得た。15KでのPL測定では3.469eVのI_2発光が支配的であり、これは残留歪みを含む場合でのI_2発光3.482eVよりも低エネルギー側であった。

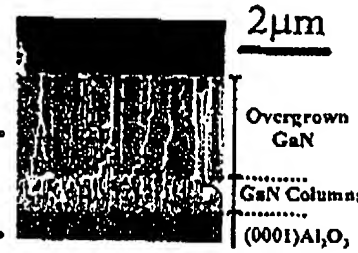

Fig.1 SEM image showing sample cross section.

謝辞　本研究は日本学術振興会未来開拓学術研究推進事業JSPS-RFTF97P00102及び一部文部省科学研究補助金#11750296の援助を受けた。
X線装置を提供していただいた東工大伊賀・小山・宮本研究室の関係者に感謝する。

文献　[1]M.Yoshizawa *et al.*, JJAP 36 L459(1997).

5a－Y－11

$Al_xGa_{1-x}N$/GaNヘテロ構造の臨界膜厚の不純物濃度依存性

Impurity concentration dependence of critical thickness of $Al_xGa_{1-x}N$/GaN heterostructure

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1. はじめに

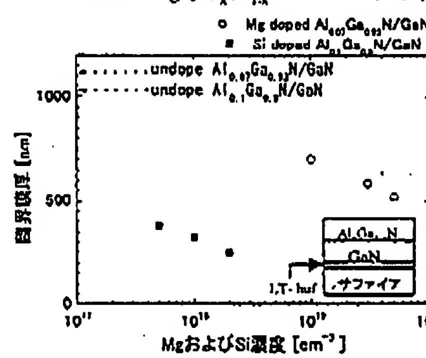
前回、我々は成長中その場観察により$Al_xGa_{1-x}N$/GaNヘテロ構造において、Si濃度の増加によりクラック発生の臨界膜厚が減少することを報告した。Mgはデバイスを作製する上で必要不可欠であるアクセプタ不純物であり、Si同様、Mgを$Al_xGa_{1-x}N$にドープした時の臨界膜厚の変化を知ることはデバイス作製上大変重要である。

2. 実験

MOVPE法によりサファイア(0001)基板上に低温緩衝層を介して成長させた単結晶アンドープGaN上に、Mgをドープした$Al_{0.07}Ga_{0.93}N$混晶を直接成長した。成長中の応力は、MOSS[1]により測定を行った。応力が緩和した時の膜厚を臨界膜厚とし、Mgの原料の供給量を変化させたときの臨界膜厚の変化を求め、Siドープの場合と比較検討した。

3. 結果

右図にGaN上に成長したMgをドープした$Al_{0.07}Ga_{0.93}N$およびSiをドープした$Al_{0.1}Ga_{0.9}N$の臨界膜厚の不純物濃度依存性を示す。Siをドープした時と同様に、Mg濃度が増加するにしたがって臨界膜厚が減少することが分かる。当日はSiとの比較などを述べ、不純物による臨界膜厚および膜に働く応力の変化について検討する。



不純物濃度と臨界膜厚の関係

参考文献　[1] J. Floro et al. J. Elect. Mat. 26 (1997) 969.

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高速成長低温AlGaNバッファ層を用いたGaN膜の高品質化

High quality GaN film on low-temperature AlGaN buffer layer grown with high growth rate

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SANYO Electric Co.,Ltd.　T.Kano, H.Ohbo, M.Hata, T.Kunisato, T.Yamaguchi, T.Goto, N.Hayashi, M.Shono, M.Sawada
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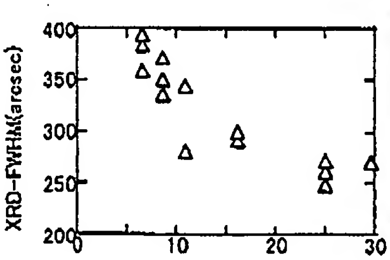
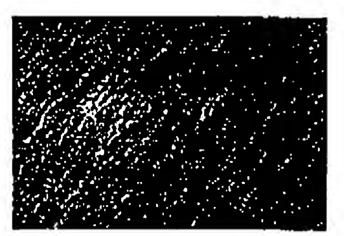
1.はじめに　サファイア上のGaN層は通常低温成長バッファ層上に成長が行われており、窒化物系発光素子の特性改善にはバッファ層およびその上に成長するGaN層の条件最適化が重要である。今回、我々はバッファ層の成長速度を著しく増加させることにより高品質GaN膜が得られることを見い出したので報告する。

2.実験　常圧MOCVD法を用いてc面サファイア上に2段階成長法でGaNの成長を行った。バッファ層はAlGaNであり、バッファ層およびその上に成長するGaN層の成長温度はそれぞれ600℃、1080℃である。バッファ層成長速度を変化させて、X線回折半値幅、表面モフォロジ等の評価を行った。

3.結果　AlGaNバッファ層の成長速度とX線半値幅の関係を図1に示す。成長速度の増加に伴い、X線回折半値幅が低減し、低温AlGaNバッファ層の成長速度が25Å/sec(9μm/h)の高速成長時に248arcsecと良





Translation of OHP sheets used in The 61st Autumn M eting, 2000
of The Japan Society of Applied Physics

High Quality GaN Film on Low-temperature AlGaN Buffer Layer Grown with High Growth Rate

Sanyo Electric Co., Ltd.
Microelectronics Research Center

Takashi Kano, Hiroki Ohbo, Masayuki Hata,
Tatsuya Kunisato, Tsutomu Yamaguchi,
Takenori Goto, Nobuhiko Hayashi,
Masayuki Shono, Minoru Sawada

Summary of Report

1. Background

2. Experimental Conditions

3. Evaluation of AlGaN Low-Temperature Buffer Layer Depending on Variation of Growth Rate
   - ·X-Ray Diffraction
   - ·Etch Pit Density
   - ·Sectional TEM

4. Characteristics of Blue Semiconductor Laser Employing High-Quality GaN Growth

5. Conclusion

Background

Conventional Low-Temperature Buffer Layer

No variations of characteristics with the growth rate have been examined.

Object

Extension of Optimum Condition Range in High-Quality GaN Growth

1. Employment of AlGaN Low-Temperature Buffer Layer
2. Quality Improvement of GaN Layer by Growth Rate Control

Growth Conditions

1. Structure of MOCVD Apparatus

1-1. Trilaminar Horizontal MOCVD Apparatus

1-2. Heating System by High-Frequency Oscillation

2. Growth Conditions for AlGaN Low-Temperature Buffer Layer

2-1. Substrate: Sapphire C-Plane Substrate

2-2. Used Materials: TMAl, TMGa, NH3, H2 and N2

$TMAl/(TMAl + TMGa) \fallingdotseq 0.5$

2-3. Growth Temperature: 600°C

2-4. Thickness of Grown Film: 120 to 140 Å

3. Growth Conditions for GaN Layer

3-2. Used Materials: TMGa, NH3, H2 and N2

3-2. Growth Temperature: 1080°C

Structure of and Method of Evaluation for Evaluated Sample

Structure of Evaluated Sample

GaN Layer (4 μm)
AlGaN Low-Temperature Buffer Layer (120 to 140 Å)
Sapphire C-Plane Substrate

Evaluation Method

1. Full Width at Half Maximum in X-Ray Diffraction Rocking Curve

   GaN(0002) Diffraction

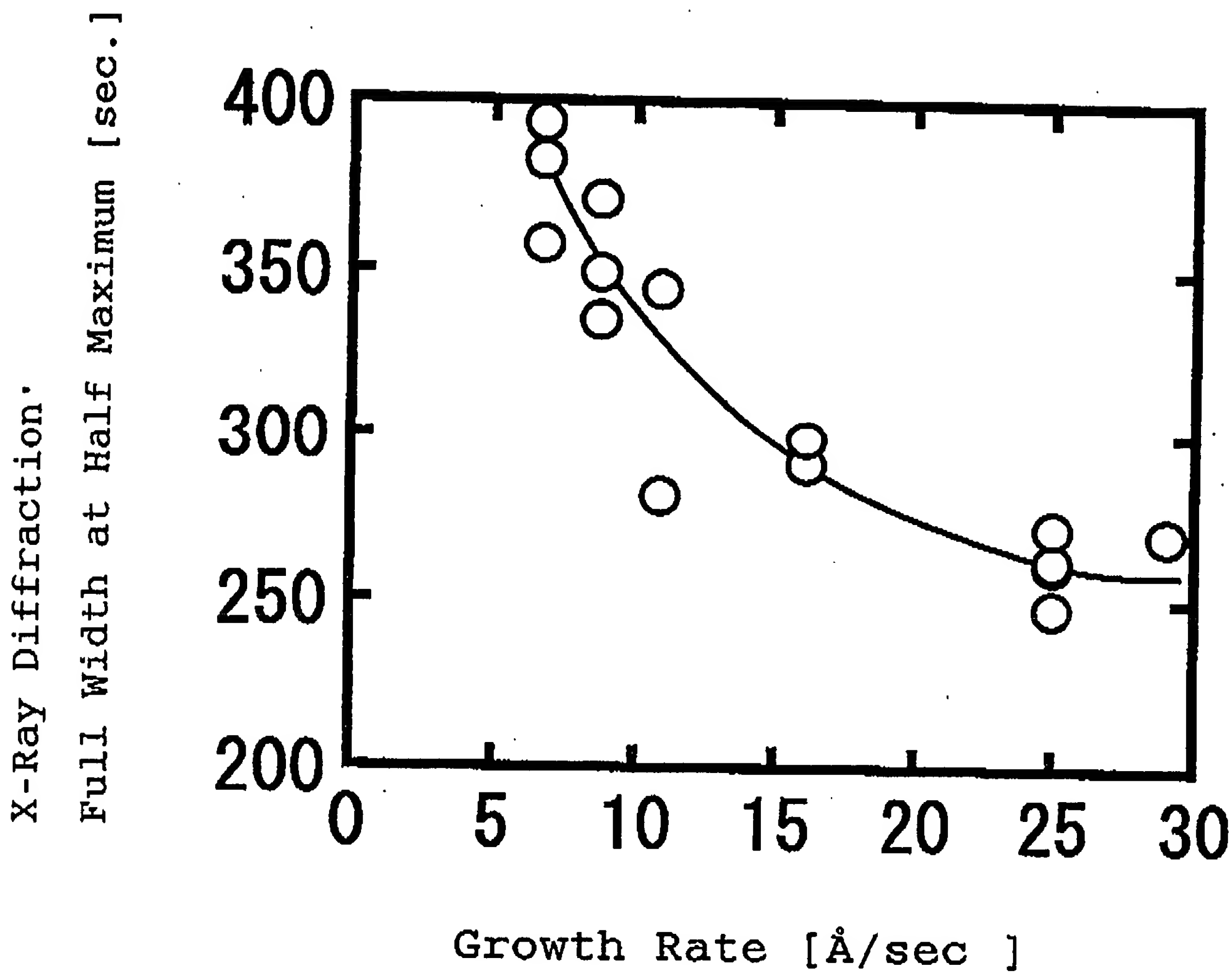
2. Etch Pit Density

   Etching Method NaOH:KOH = 5:1 (280°C)(1)

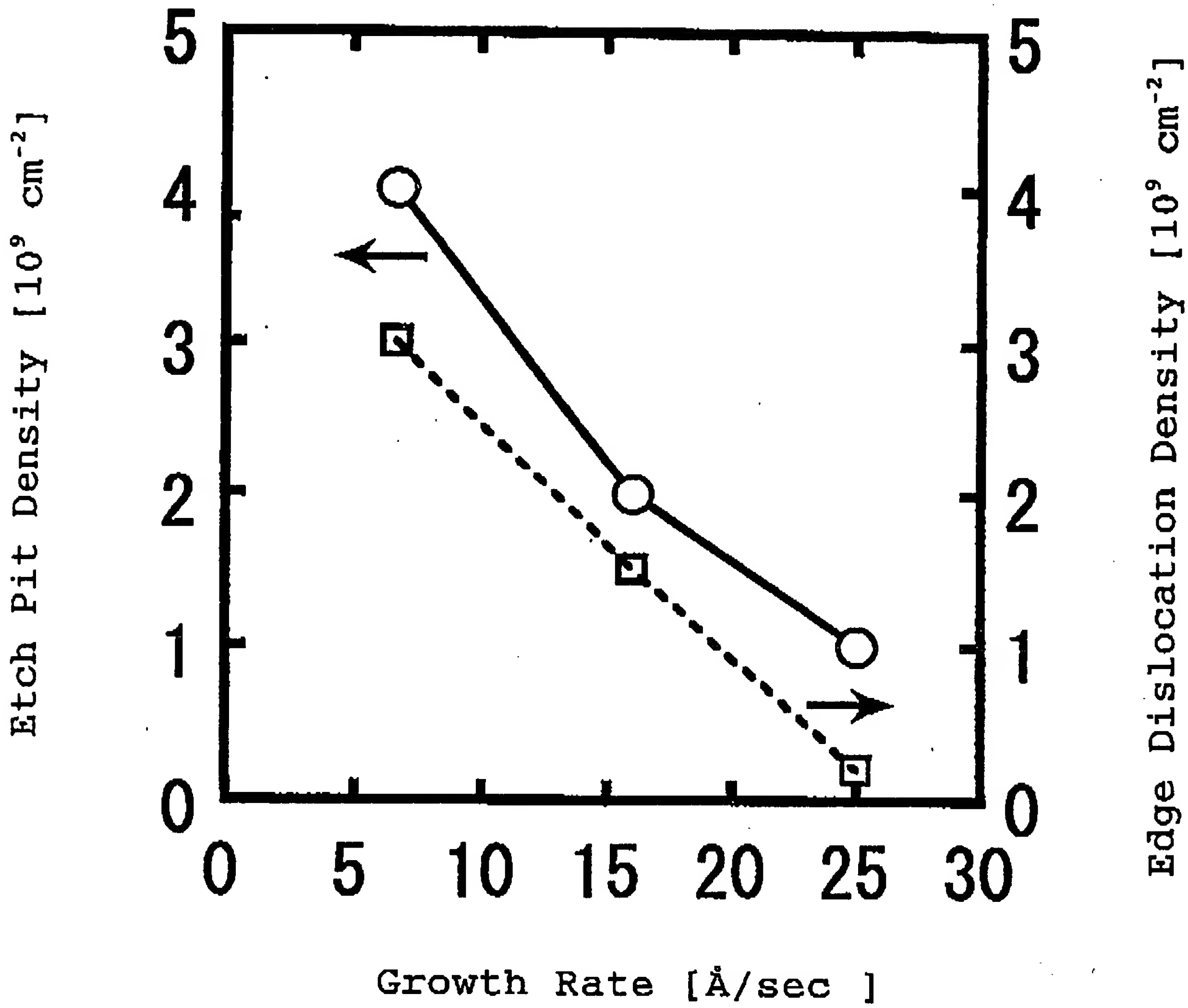
3. Sectional TEM Observation

(1) "Evaluation of Defects by Etch Pit in GaN " by Masayuki Hata et. al., Sanyo Electric Co., Ltd. Microelectronics Research Center

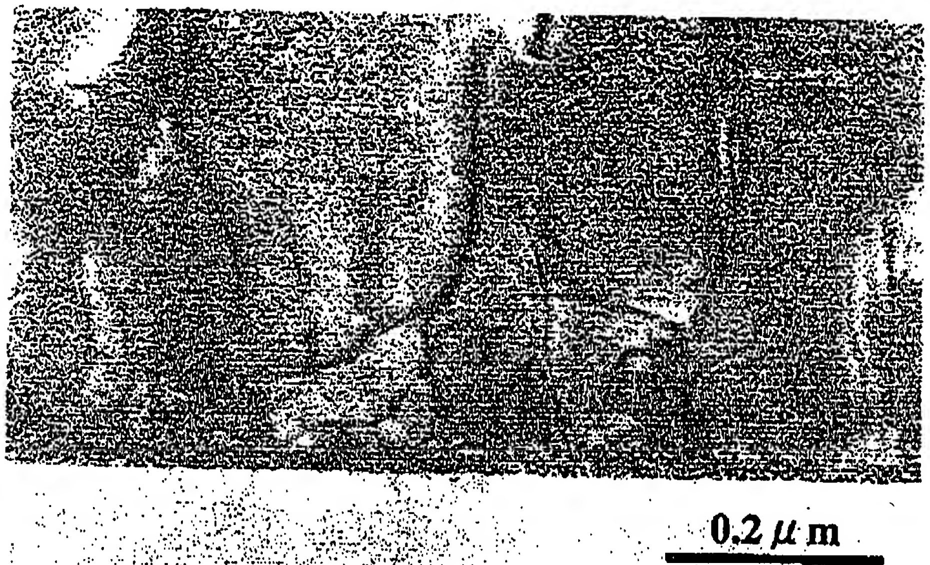
Extended Abstracts of th 57th Meeting of the Japan Society of Applied Physics (1996), No. 1, p. 302



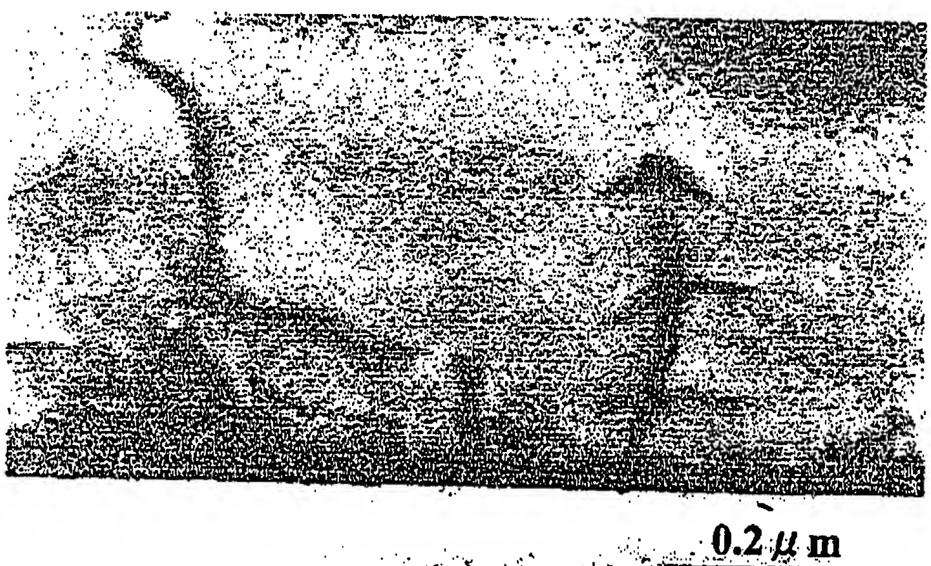
Relation Between Growth Rate of AlGaN Low-Temperature Buffer Layer and Full Width at Half Maximum of X-Ray of GaN Layer



Relation Between Growth Rate of AlGaN Low-Temperature Buffer Layer and Etch Pit Density of GaN Layer

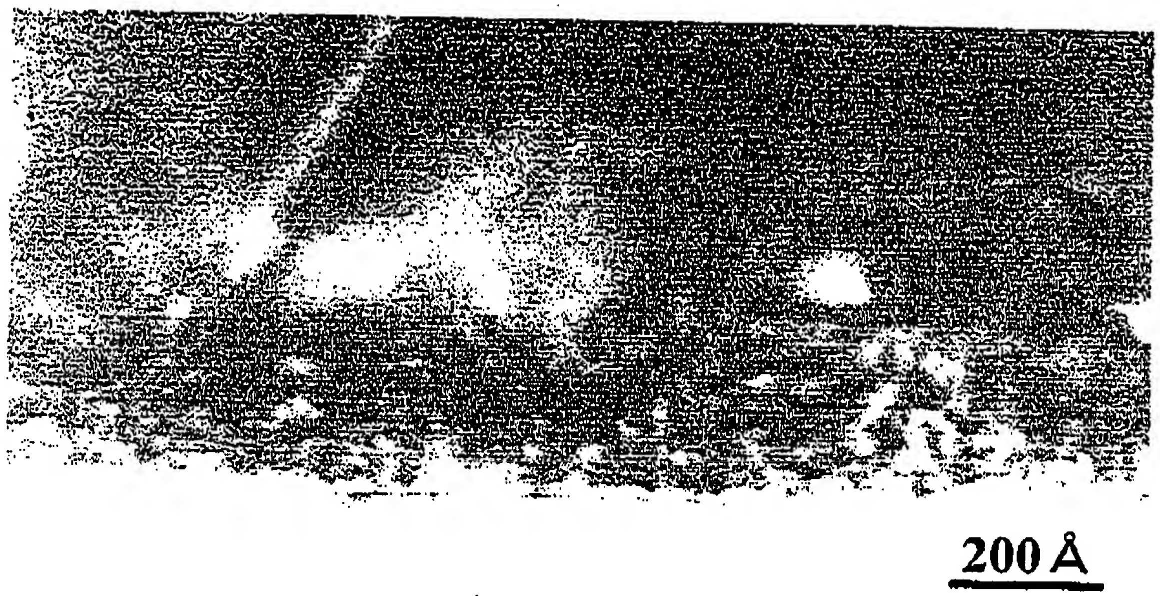


Growth Rate: 6.7 Å/sec.

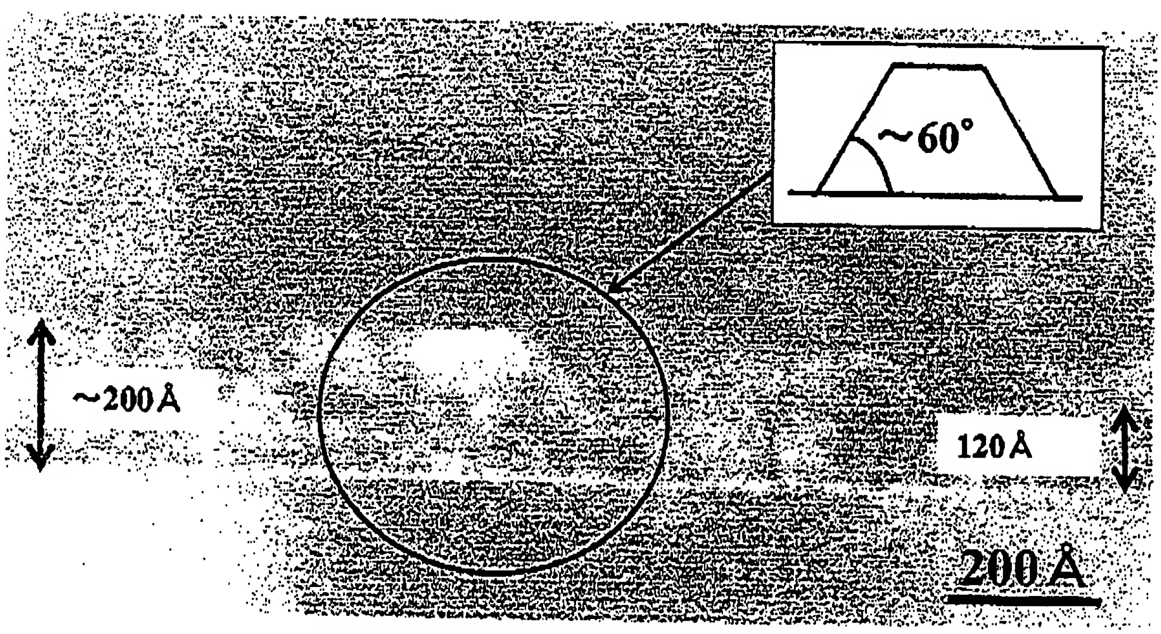


Growth Rate: 25.0 Å/sec.

Sectional TEM Photograph of Interface Between Sapphire Substrate and GaN Layer (× 300,000)
[Sectional Photograph on GaN (11-20) Plane]



Growth Rate: 6.7 Å/sec.



Growth Rate: 25.0 Å/sec.

Sectional TEM Photograph of Interface Between Sapphire Substrate and GaN Layer (×2,000,000)
[Sectional Photograph on GaN (11-20) Plane]

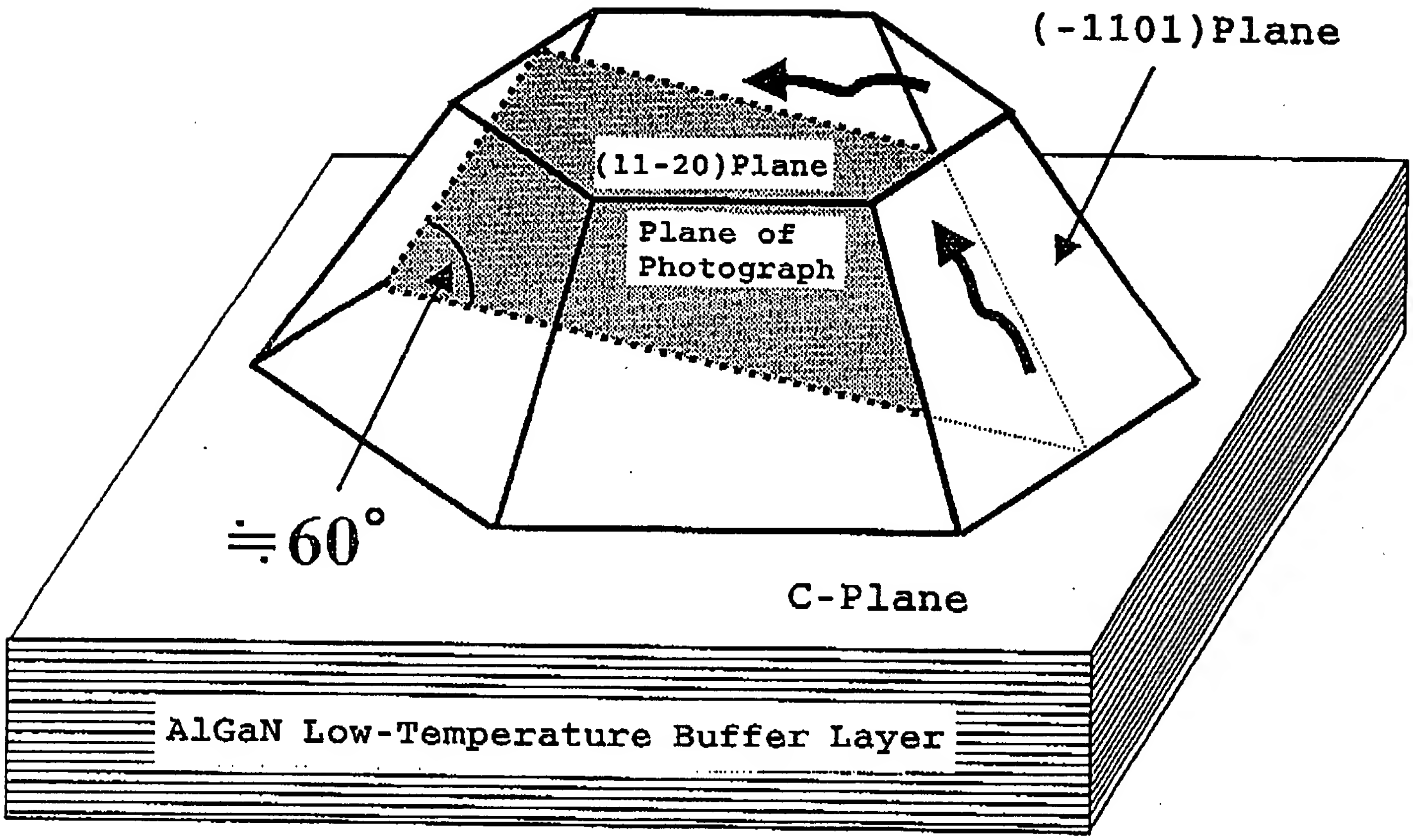
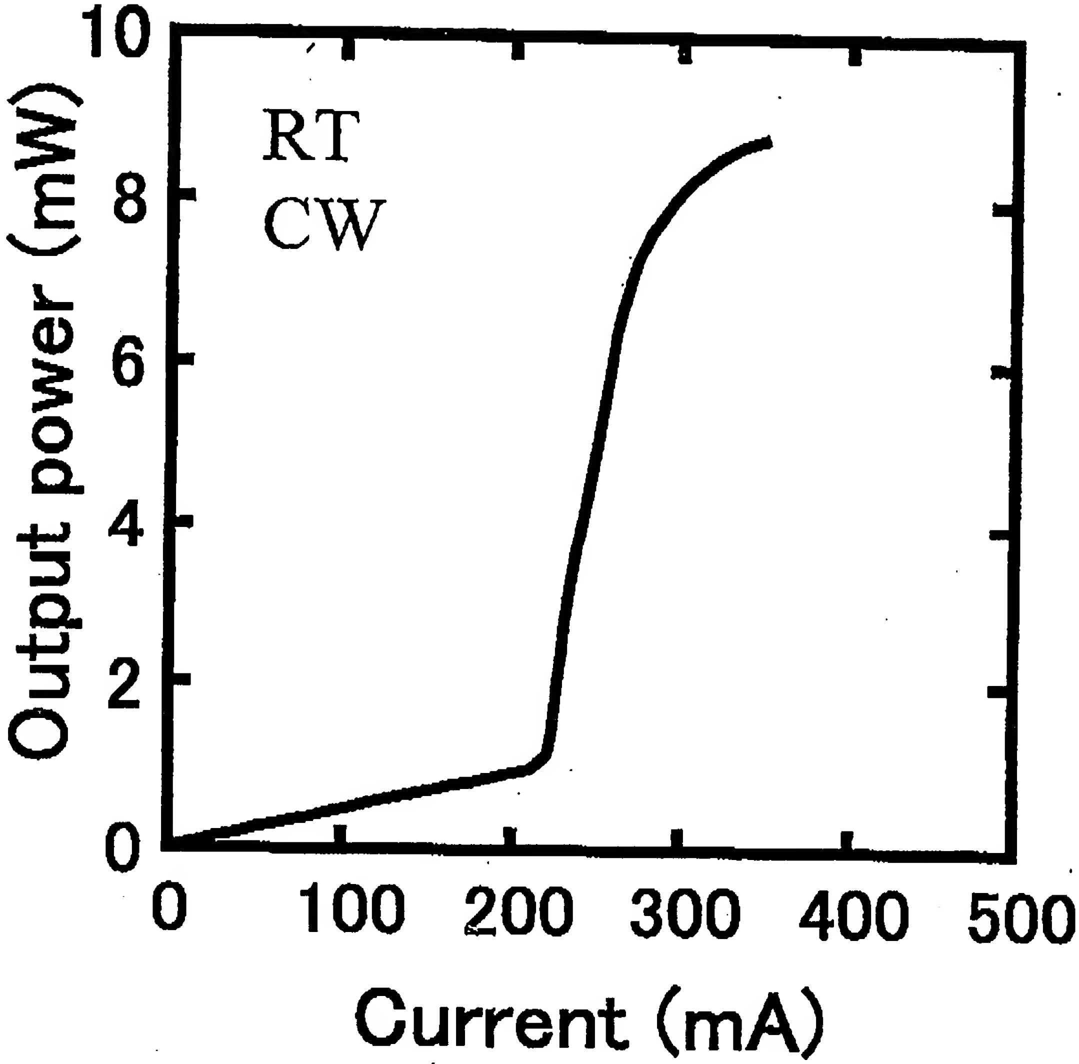


Image Diagram of Direction of Defect in Initial State of Growth of GaN Layer Employing Fast-Grown AlGaN Low-Temperature Buffer Layer



I-L Characteristics of GaN Laser Employing High-Quality GaN Growth

(Room-Temperature Continuous Oscillation)

Conclusion

1. Increasing growth rate of AlGaN low-temperatur buffer layer to 25 to 30 Å/sec.

GaN Layer

・Full Width at Half Maximum of X-Ray Rocking Curve: 250 sec.

・Etch Pit Density: 1.0 × [10^9 cm^{-2}]

↓

From sectional TEM on the interface between sapphire and GaN:

① Most of defects caused on the interface progress in directions parallel to the (-1101) plane and the C-plane.

↓

② The number of through defects in the C-axis direction decreases.

2. A blue semiconductor laser of room-temperature continuous oscillation was obtained through high-quality GaN growth.

高速成長低温AlGaNバッファ層を用いたGaN膜の高品質化

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國里竜也　山口勤　後藤壮謙
林伸彦　庄野昌幸　澤田 稔

発表の概要

1.背景

2.実験条件

3. AlGaN低温バッファ層の成長速度変化による評価
   - X線回折
   - エッチ・ピット密度
   - 断面TEM

4.高品質GaN成長を用いた青色半導体レーザの特性

5.まとめ

背景

これまでの低温バッファ層

成長速度による特性の変化は
調べられていない。

目的

高品質GaN成長における最適条件範囲の拡大

1.AlGaN低温バッファ層の採用
2.成長速度制御によるGaN層の
高品質化

成長条件

1.MOCVD装置構成

1-1.三層流横形MOCVD装置

1-2.高周波発振による加熱方式

2.AlGaN低温バッファ層の成長条件

2-1.基板:　サファイアC面基板

2-2.使用原料:TMAl,TMGa, NH3, H2,N2

TMAl/(TMAl+TMGa)≒0.5

2-3.成長温度:600℃

2-4.成長膜厚:120～140Å

3.GaN層の成長条件

3-2.使用原料:TMGa, NH3, H2,N2

3-2.成長温度:1080℃

評価サンプルの構造と評価方法

評価サンプルの構造

GaN層（4 μm）
AlGaN低温バッファ層（120～140 Å）
サファイアC面基板

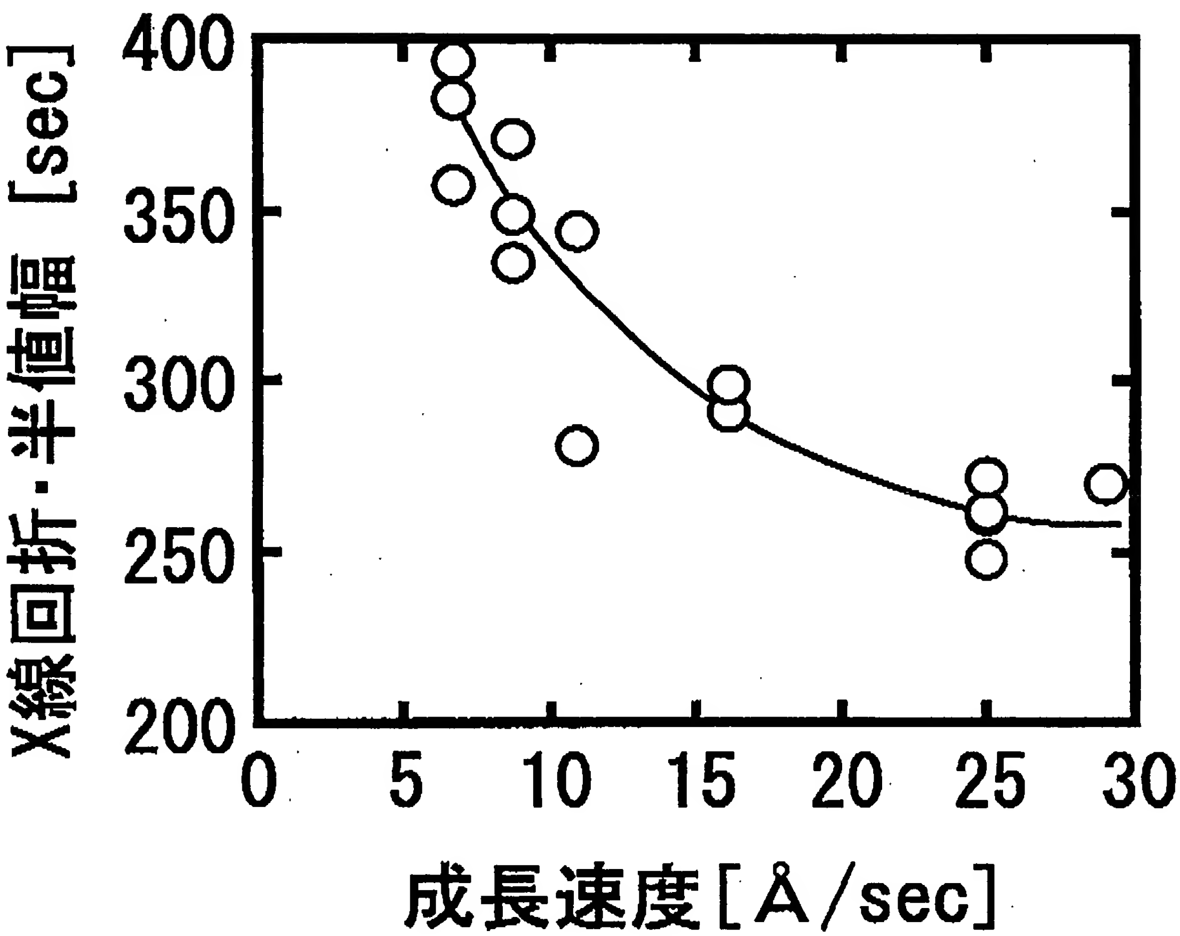
評価方法

1.X線回折ロッキングカーブ半値幅
GaN(0002)回折

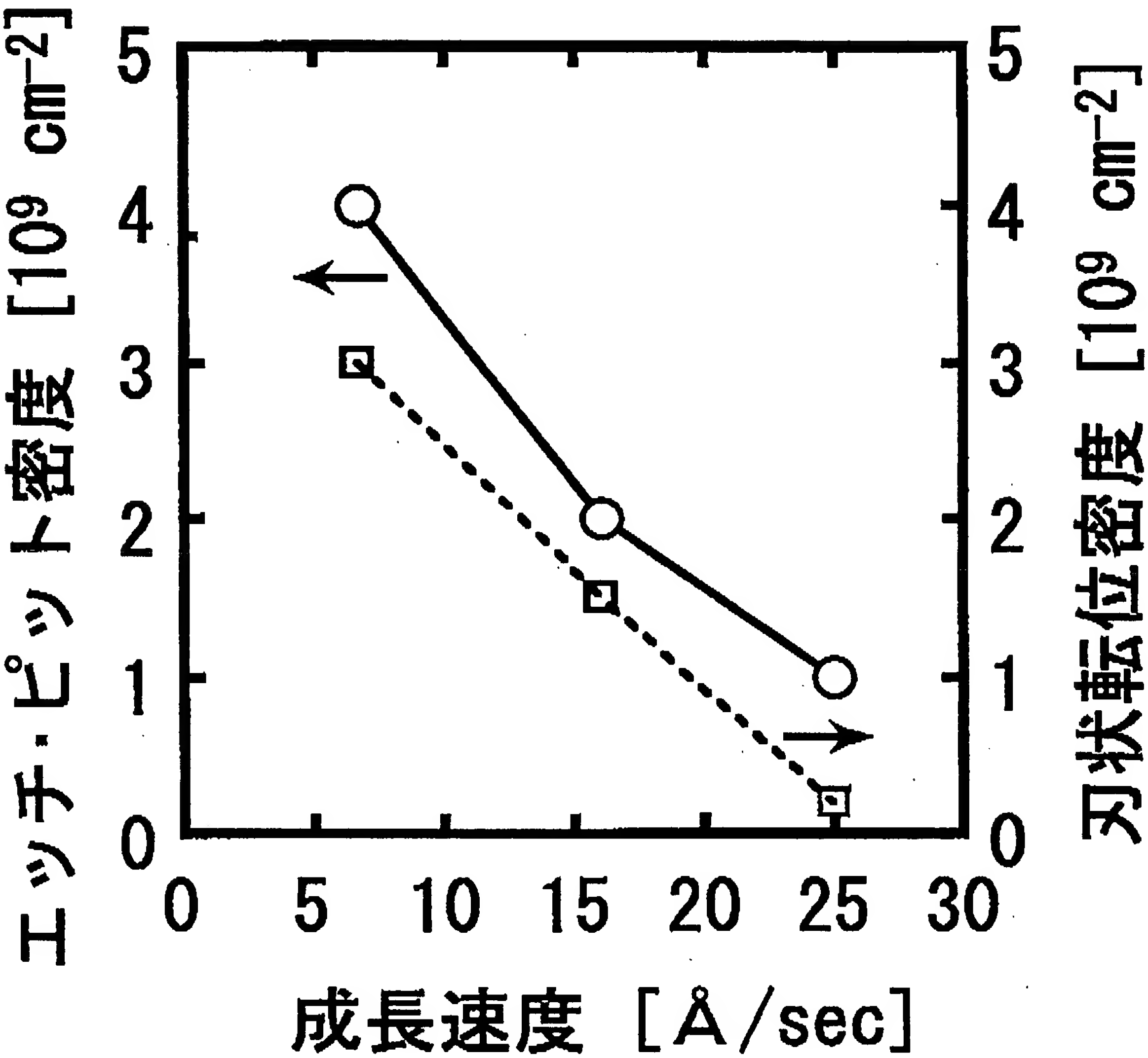
2.エッチ・ピット密度
エッチング方法 NaOH:KOH=5:1 (280℃)[1]

3.断面TEM観察

(1)畑雅幸 et. all 三洋電機（株）マイクロエレクトロニクス研究所
第57回応用物理学会学術講演会 予稿集 (1996) No.1, p302
「エッチピットによるGaNの欠陥の評価」

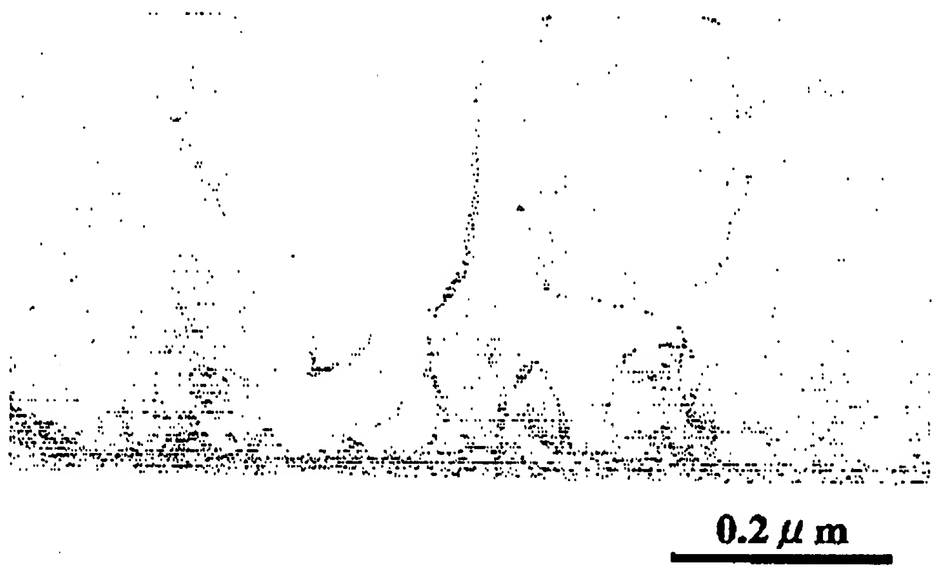


AlGaN低温バッファ層の成長速度とGaN層のX線半値幅の関係

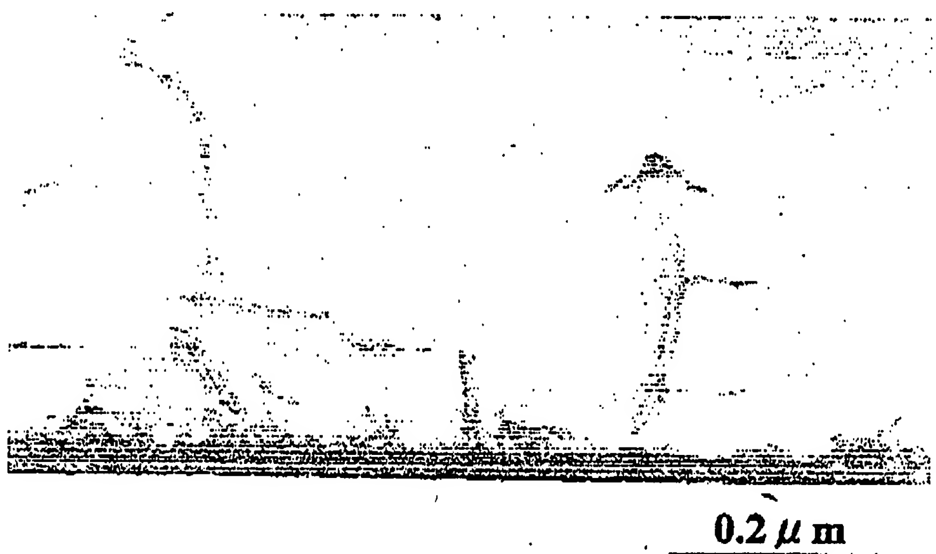


AlGaN低温バッファ層の成長速度とGaN層のエッチ・ピット密度の関係

2



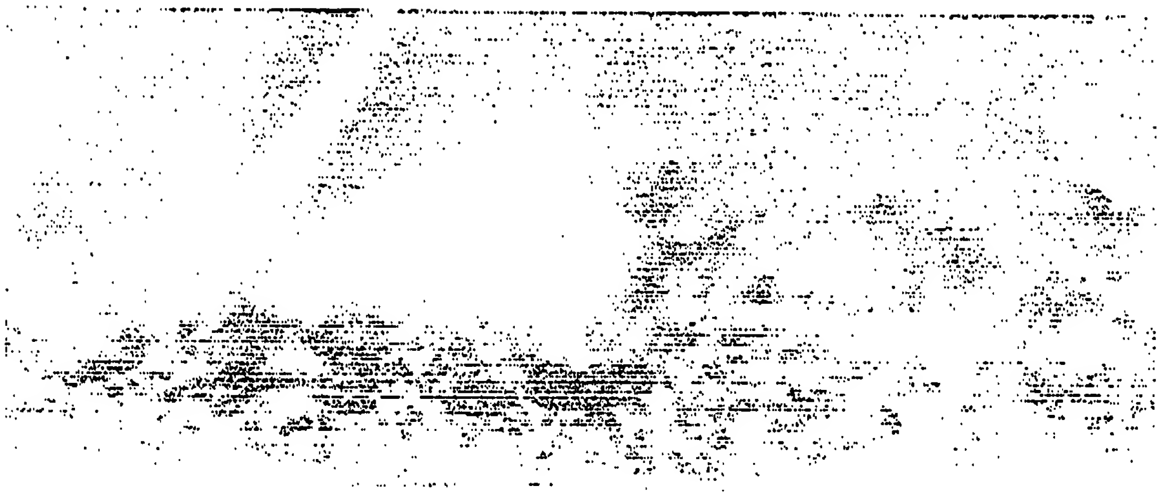
成長速度:6.7Å/sec



成長速度:25.0Å/sec

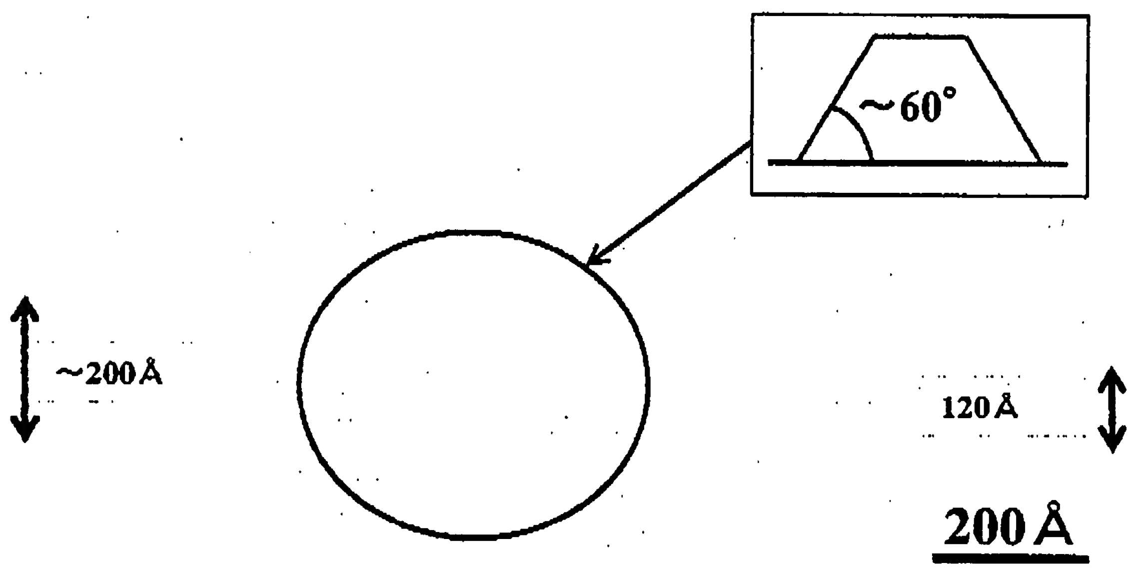
サファイア基板とGaN層の界面の
断面TEM写真　　　　　(×300,000)
[GaN(11-20)面での断面写真]

①



200 Å

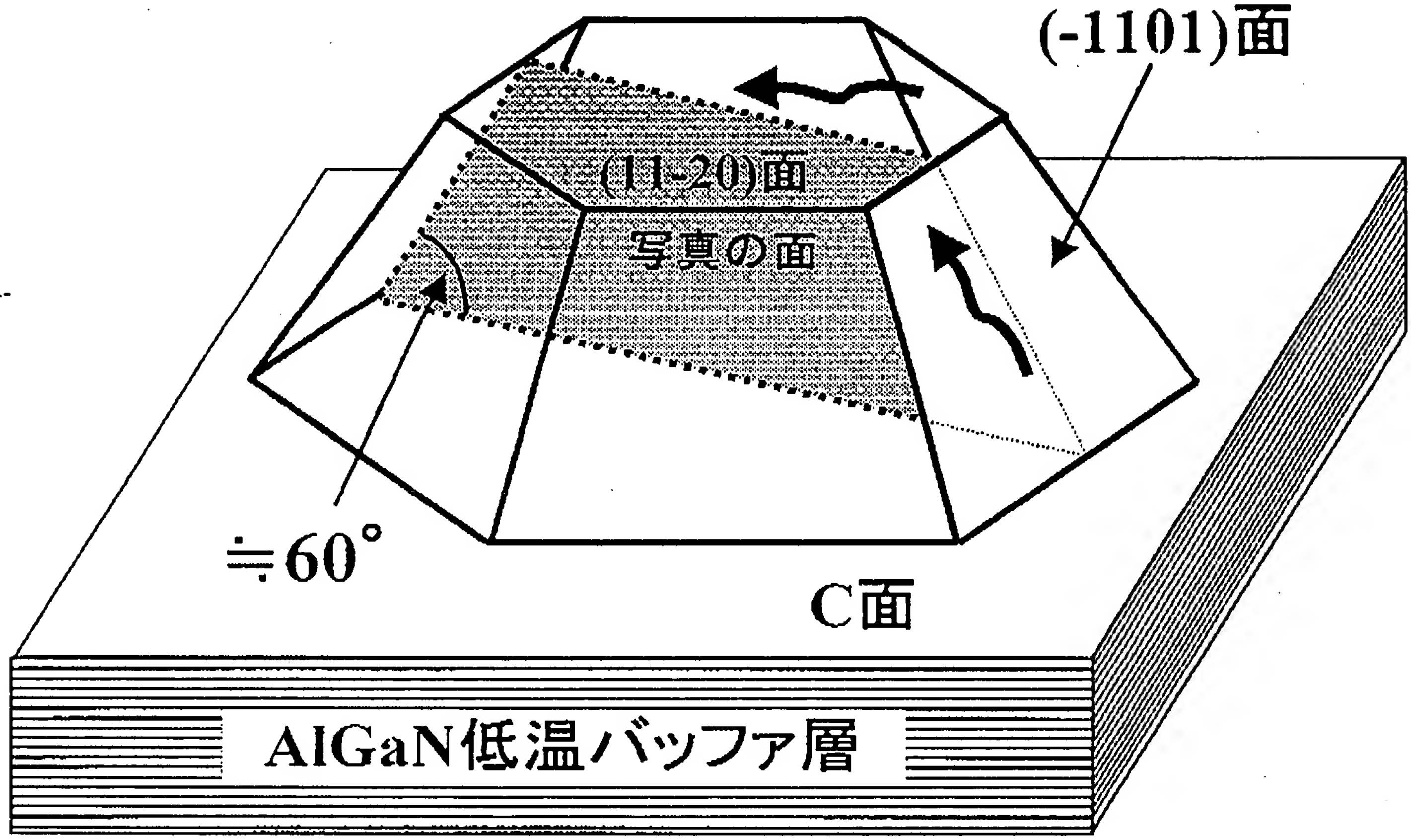
成長速度: 6.7 Å/sec



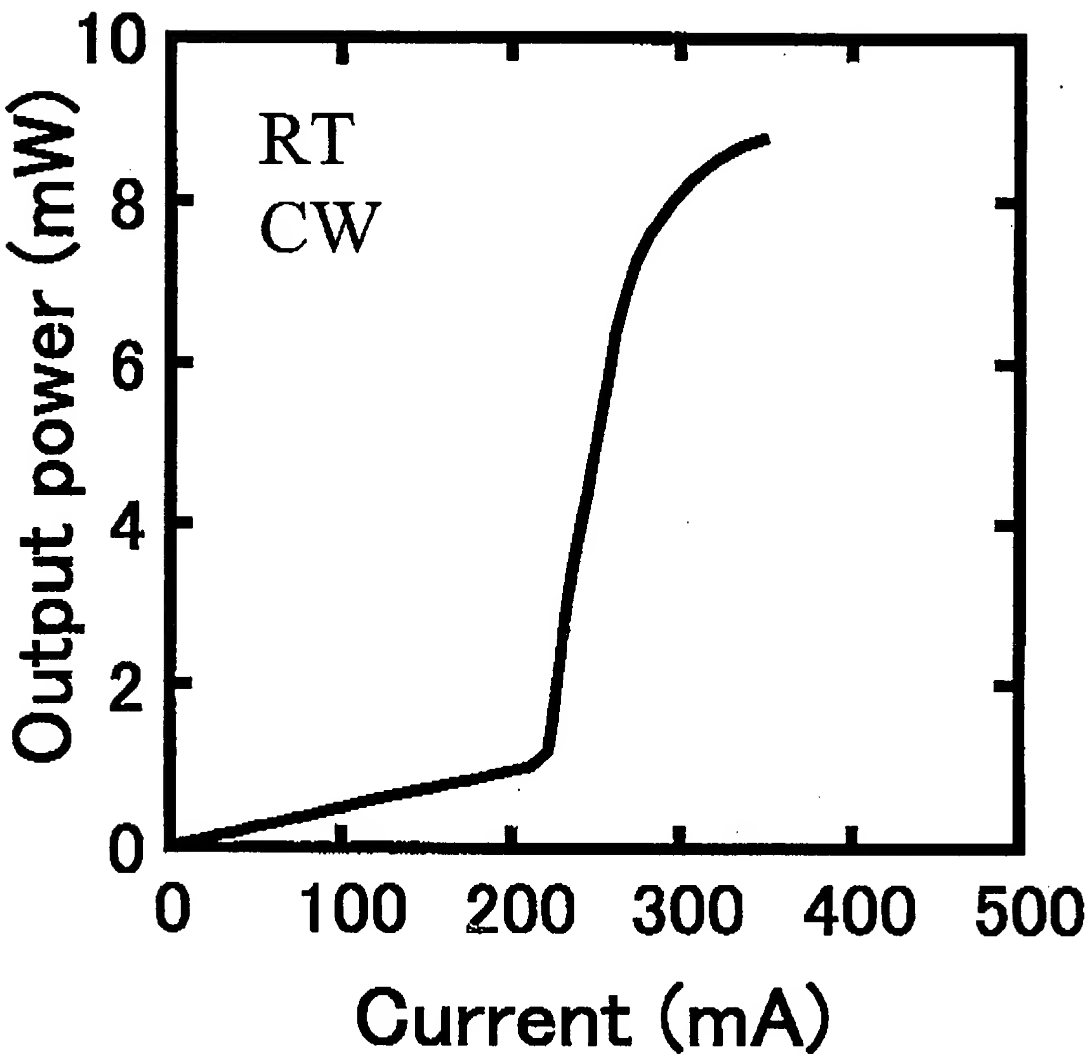
成長速度: 25 Å/sec

サファイア基板とGaN層の界面の
断面TEM写真　　　(×2, 000, 000)
[GaN(11-20)面での断面写真]

③



高速成長AlGaN低温バッファ層を用いた
成長初期のGaN層での欠陥の方向
のイメージ図



高品質GaN成長を用いた
GaNレーザのI-L特性
(室温連続発振)

まとめ

1.AlGaN低温バッファ層の成長速度を 25〜30Å/secにまで増加

GaN層

・X線ロッキングカーブ半値幅：250sec
・エッチ・ピット密度：1.0×10^9 [cm-2]

↓

サファイアとGaN界面の断面TEMより

①界面で発生した欠陥の多くが(−1101)面、C面 と平行な方向に進んでいる。

↓

②C軸方向の貫通転位が減少する。

2.高品質GaN成長を用いて室温連続発振の青色半導体レーザを得た。